[1] B. Rohrmoser, G. Eickerling, M. Presnitz, W. Scherer, V. Eyert, R.-D. Hoffmann, U. Ch. Rodewald, Ch. Vogt, R. Poettgen, J. Am. Chem. Soc. (2007), 129, 9356-9365.

Keywords: charge density studies, topological properties of charge distribution, quantum chemistry

MS.75.2

Acta Cryst. (2008). A**64**, C128

Study of electronic structure of tetrakis(μ_2-Acetato)-diaqua-di-copper(II) complex

Jozef Kozisek, Lucia Perasinova, Martin Breza
Slovak University of Technology in Bratislava, Physical Chemistry, Radlinskeho 9, Bratislava, Slovak Republic, SK 812 37, Slovak Republic, E-mail：jozef.kozisek@stuba.sk

The nature of Cu-Cu interaction in tetrakis(μ_2-Acetato)-diaqua-di-copper(ii) complex has been studied by both experimental and theoretical treatments. A large experimental data set (CCD GEMINI R diffractometer, 387268 diffractions at 100 K, resolution of 0.39 Å, an average redundancy of 30.6) was measured. The data reduction (*CrysAlis* [1]) gives a unique 12674 diffractions (R_{Int} = 0.026, R(σ) = 0.006). Refinement with the XD package [2] gives R{ F } = 0.0187. In order to identify the systematic errors in the experimental data sets of structure factors, the new procedure for obtaining the theoretical structure factors at 100 K from the theoretical grid electron density has been developed. The electron density at grid points is evaluated by CRYSTAL06 software for periodic quantum-chemical calculations at B3LYP level of theory [3]. The distance between the discrete grid point and the closest particular atom is used as a criterion for assigning the temperature factor to each grid point. The procedure developed might enable us to obtain spin density distribution, too.
This work has been supported by Slovak Grant Agency VEGA (project No. 1/0817/08)
[1] Oxford Diffraction (2008). CrysAlis RED 171.32.15. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
[2] R. Dovesi *et al.*; CRYSTAL06 User's Manual, University of Torino, Torino, 2006.
[3] T. Koritsanszky *et al.*; XD, A Computer Program Package for Multipole Refinement and Analysis of Charge Densities from X-ray Diffraction Data. Freie Universität Berlin. User Manual (2003).

Keywords: charge density, spin density, magnetic properties

MS.75.3

Acta Cryst. (2008). A**64**, C128

Estimation of optical properties from wavefunction fitting of X-ray diffraction data

Parthapratim Munshi[1], Birger Dittrich[2], Mark A Spackman[1], Dylan Jayatilaka[1], Leigh H Rees[3]
[1]The University of Western Australia, Chemistry, Chemistry M313, 35 Stirling Highway, Perth, Crawley, WA, 6009, Australia, [2]Universitat Gottingen Institut fur Anorganische Chemie, Tammannstrabe 4, D-37077 Gottingen, Germany, [3]Oxford Diffraction Ltd, 8 Milton Park, Abingdon, Oxfordshire OX14 4RX, UK, E-mail：munshi@cyllene.uwa.edu.au

In general, the crystallography of important nonlinear optical (NLO) materials is not well described and much remains to be done to characterize their relevant properties (electric and optical) in the solid state. This encouraged us to perform the detailed charge density studies on a series of organic molecular crystals with known NLO properties. The materials of interest are 2-(N-prolinol)-5-nitropyridine (PNP), N-(4-nitrophenyl)-L-prolinol (NPP) and 3-methyl 4-nitropyridine N-oxide (POM), which have very high second order NLO coefficients. Charge density analyses have already been reported for NPP [1] and POM [2], but we are revisiting these materials, along with PNP, to critically test a number of novel approaches to the estimation of linear and nonlinear optical properties using constrained wavefunctions fitted to the X-ray diffraction data [3]. Charge density analyses are based on X-ray diffraction data collected on an Oxford Diffraction Xcalibur S instrument at 100 K. Hydrogen atom ADPs are estimated using a recently described SHADE2 procedure [4]. Results presented will include conventional multipole refinements, details of wavefunction fitting, estimates of the zero-frequency dipole polarisability tensors for the molecules and crystal refractive indices, and molecular first hyperpolarisability tensors. Critical comparison of the estimated results with independent experimental data will be made where possible.
[1] Fkyerat, A. et al., Acta Cryst., 1995, B51, 197; Phys. Rev., 1996, B53, 16236.
[2] Zyss, J. et al., J. Chem. Phys., 1981, 74(9), 4800 - 4811.
[3] Jayatilaka, D. & Grimwood, D. J., Acta Cryst., 2004, A60, 111 - 119 and references there in.
[4] Munshi, P. et al., Acta Cryst., 2008, A, submitted.

Keywords: charge density, optical properties of crystals, wavefunction fitting

MS.75.4

Acta Cryst. (2008). A**64**, C128–129

Magnetic interactions in thiazyl-based magnets: The role of the charge and spin densities

Javier Campo[1], Javier Luzon[1,2], Fernando Palacio[1], Garry J McIntyre[2], Jeremy M Rawson[3], Antonio J Alberola[3], Andres E Goeta[4]
[1]Materials Science Institute of Aragon, Low Temperature Physics, Facultad de Ciencias, C/ Pedro Cerbuna 12, Zaragoza, Spain, 50009, Spain, [2]Institute Laue Langevin, 6 Rue Jules Horowitz, Grenoble, 38042, France, [3]Department of Chemistry, The University of Cambridge, Lensfield Road, Cambridge CB2 1EW, UK, [4]Department of Chemistry, University of Durham, South Road, Durham DH1 3LE, UK, E-mail：jcampo@unizar.es

The crystal structure of the organic radical p-$O_2NC_6F_4CNSSN$ was determined at 20 K through a single-crystal neutron-diffraction experiment. It crystallises in the tetragonal space group $P4_12_12$, unchanged from a previous single-crystal X-ray diffraction experiment at 220 K although there are some changes in molecular geometry and intermolecular contacts arising from the contraction of the unit cell. Polarized neutron diffraction at 1.5 K revealed that the spin distribution is predominantly localised on the N and S atoms of the heterocyclic ring with a small negative spin density on the heterocyclic C atom. Spin populations determined using a multipolar analysis were -0.06, +0.25 and +0.28 on the C, N and S sites, respectively. These spin populations are in excellent agreement with both ab-initio DFT calculations (spin populations on the C, N and S sites of -0.07, 0.22 and 0.31, respectively) and cw-EPR studies which estimated the spin population on the N site as 0.24. The DFT calculated spin density revealed less than 1% spin delocalisation onto the perfluoroaryl ring, several orders of magnitude lower than the density on the heterocyclic ring. cw-ENDOR studies at both X-band (9 GHz) and Q-band (34 GHz) frequencies probed the spin populations at the two chemically distinct F atoms. These spin populations on the F atoms ortho and meta to the dithiadiazolyl ring are of magnitude 10-3 and 10-4 respectively. Additional high-

resolution single-crystal X-ray diffraction studies at 100 K analysed within the atoms-in-molecules (AIM) framework gave detailed information on the charge density distribution.

Keywords: dithiadiazolyl, organic Ferromagnet, spin density

MS.75.5

Acta Cryst. (2008). A**64**, C129

Observation of spin densities by the X-ray magnetic diffraction

Masahisa Ito
Gunma University, Graduate School of Engineering, Tenjin-cho 1-5-1, Kiryu, Gunma, 376-8515, Japan, E-mail：itom@phys.sci.gunma-u.ac.jp

Observation of density distribution of magnetic moment in ferromagnets has been performed solely by the neutron magnetic diffraction which utilizes interaction between the neutron spin and the magnetic moments. Recently intense elliptically-polarized X-rays of synchrotron radiation have come to be available, and the observation of magnetic moment is becoming possible by the X-ray magnetic diffraction (XMD) which utilizes interaction between the photon helicity and the magnetic moment through pioneering researches [1-4]. The XMD, that is nonresonant X-ray magnetic Bragg scattering, is a peculiar experimental method which enables us to measure separately spin and orbital component of the magnetic moments of ferromagnets. Physical quantity directly observed by this method is spin and orbital component (or mixture of them) of the magnetic form factor. By inverse Fourier transform of the spin or orbital magnetic form factor, the real-space density distribution of spin or orbital magnetic moment can be obtained. We have constructed an XMD experimental system on the BL3C of KEK-PF at Tsukuba in Japan, and have been performing the XMD experiments. In this lecture three dimensional spin density of an orbital-ordering perovskite $YTiO_3$ obtained by the XMD is shown, which reproduced the electron distribution of 3d-t2g electrons of Ti atoms. This result would prove that the XMD method is a useful tool to observe spin density of ferromagnets.
[1] M. Brunel et al., Acta Crystallogr. A39 84 (1983).
[2] M. Blume, J. Appl. Phys. 57 3615 (1985), and, M. Blume and D. Gibbs, Phys. Rev. B37 1779 (1988).
[3] S. W. Lovesey, J. Phys. C20, 5625 (1987).
[4] D. Laundy et al., J. Phys. :Condens. Matter 3 369 (1991), and, S. P. Collins et al., Philos. Mag. B65 37 (1992).

Keywords: X-ray magnetic scattering, spin density, titanates

MS.76.1

Acta Cryst. (2008). A**64**, C129

Coherent diffractive imaging: A new tool for high resolution X-ray imaging

Keith A Nugent[1], Garth J Williams[1], Brian Abbey[1], Andrew G Peele[2], Mark Pfeifer[2], Jesse N Clark[2], Martin De Jonge[3], Ian McNulty[4]
[1]The University of Melbourne, School of Physics, Tin Alley, The University of Melbourne, Vic, 3010, Australia, [2]La Trobe University, Bundoora, Vic., 3086, Australia, [3]Australian Synchrotron, 800 Blackburn Road, Clayton, Vic., 3168, [4]Advanced photon Source, Argonne National Laboratory, 9700 S. Cass Avenue, Argonne, Ill., 60439, USA, E-mail：keithan@unimelb.edu.au

The increased availability of high coherence X-ray sources, such as 3rd generation synchrotrons and more recently X-ray free electron lasers1, has driven the development of many new forms of microscopy. Among these techniques coherent diffractive imaging (CDI) is one of the most promising, offering very high-resolution lensless imaging of non-crystallographic samples. The method computationally derives an image of a sample from a single measurement of its diffraction pattern. Image reconstruction has hitherto only been possible for isolated samples fully contained within the illuminating beam, presenting what has been seen as a fundamental limitation on the CDI method. We demonstrate here a form of CDI that can image objects of arbitrary size and which can be used to create a well-defined field of view within a complex environment. A diverging beam created by a focusing optic is used to define the region of interest, allowing it to be imaged within a much larger sample. We test the concept using visible light and then use x-rays and a high resolution test pattern to demonstrate a diffraction-limited spatial resolution of 16 nm. A key requirement of the method is that the wavefront have significant curvature over the region of interest and this is determined by the size of the focal spot. As x-ray focal spots approach 10 nm in size2, imaging of a single quantum dot or of a small virus located within a complex host environment will be possible. Thus, the technique lends itself to high-resolution structural imaging of samples previously inaccessible to CDI.

Keywords: imaging, diffraction, coherence

MS.76.2

Acta Cryst. (2008). A**64**, C129

Diffractive imaging and serial crystallography

Uwe J Weierstall[1], J CH Spence[1], D Starodub[1], K Schmidt[1], R B Doak[1], D Shapiro[2], H Chapman[3], S Marchesini[2]
[1]Arizona State University, Physics, University Dr, Tempe, AZ, 85287, USA, [2]Advanced Light Source, Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720, USA, [3]Centre for Free-Electron Laser Science, U. Hamburg / DESY, Notkestrasse 85, 22607 Hamburg, Germany, E-mail：weier@asu.edu

The inversion of single particle diffraction patterns offers aberration-free, diffraction-limited, three-dimensional images without the resolution and depth-of-field limitations of lens-based tomographic systems. Radiation damage becomes the main limiting factor for diffractive imaging of organic molecules. To overcome this limitation, two schemes have been proposed: femtosecond diffraction of single molecules with a FEL and serial diffraction of laser aligned molecules. In both cases the 3D charge density is reconstructed from the diffraction patterns via iterative phase retrieval algorithms. Our work on iterative phase retrieval and possible applications to serial crystallography will be reviewed.
J.C.H. Spence and U. Weierstall, Phase recovery and lensless imaging by iterative methods in optical and electron diffraction. Philosophical Transactions: Mathematical, Physical and Engineering Sciences Volume 360, Number 1794 (2002) 875-895.
J.C.H. Spence, K. Schmidt, J. Wu, G. Hembree, U. Weierstall, B. Doak, and P. Fromme, Diffraction from a beam of laser-aligned proteins: resolution limits. Acta Crystallographica A 61 (2005) 237-245.
D. Starodub, P. Rez, G. Hembree, M. Howells, D. Shapiro, H. N. Chapman, P. Fromme, K. Schmidt, U. Weierstall, R. B. Doak and J. C. H. Spence, Dose, exposure time and resolution in serial X-ray Crystallography, J. Synchrotron Rad. (2008). 15, 62-73

Keywords: diffraction methods, phasing, inverse problem